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Description

Process for Producing Microparticles and Apparatus therefor

Technical Field

[0001]

The present invention relates to a process for producing microparticles of a material such as indium oxide-tin oxide powder, and to an apparatus for producing the microparticles.

Background Art

[0002]

Sputtering is a generally known technique for forming thin film. In the sputtering technique, a thin film is formed by sputtering a sputtering target. The sputtering technique is employed in industrial processes, since a thin film of large surface area can be readily formed, and a high-performance film can be formed at high efficiency. In recent years, various sputtering techniques have been known, such as reactive sputtering; i.e., sputtering in a reactive gas, and magnetron sputtering, which realizes high-speed thin film formation by placing a magnet on the backside of a target.

[0003]

Among thin film products obtained through sputtering, indium oxide-tin oxide (In_2O_3-SnO_2 compound oxide, hereinafter abbreviated to as ITO) film is a transparent conductive film which has high optical transparency with respect to visible

light and high conductivity and which, therefore, finds a wide variety of uses such as for a liquid crystal display, a heat-generating film for defogging a glass panel, and an IR-reflecting film.

[0004]

Thus, in order to produce thin films with higher efficiency and lower cost, modification and improvement of sputtering conditions and sputtering apparatuses are required and are now under way, and effective operation of sputtering apparatuses is essential. In the production of ITO film through sputtering, the period from setting of a new sputtering target to termination of initial arc (anomalous discharge); i.e., the period required for initiating formation of thin films, is preferably as short as possible, and assessing the sputter-enabling period of a target from the setting thereof (cumulative sputtering time: target life) is a key issue.

[0005]

The aforementioned sputtering target for forming an ITO film is produced through mixing indium oxide powder and tin oxide powder at a predetermined ratio, molding under dry or wet conditions, and sintering the molded product (Patent Document 1). In this connection, highly-dispersible indium oxide powder has been proposed for producing high-density sintered ITO (see, for example, Patent Documents 2, 3, and 4).

[0006]

Another known method includes sintering an ITO powder

synthesized through the co-precipitation method under wet conditions (see, for example Patent Document 5). Similarly, a variety of wet-synthesis methods for producing ITO powder have been proposed for producing high-density sintered ITO (see, for example, Patent Documents 6 to 9).

[0007]

Yet another method for producing ITO powder has been proposed in which an indium-tin alloy is reacted with oxygen in plasma arc, followed by cooling the reaction product at a predetermined cooling rate or faster by means of a gas flow at a Mach number of ≥1 (see Patent Document 10). However, use of a high-speed gas flow at a Mach number of ≥1 requires large facilities, problematically impeding production of ITO powder at low cost and with high efficiency.

[0008]

Besides the ITO powder production methods, the following methods for producing metal oxide microparticles have been proposed. For example, there have been proposed a variety of methods including feeding a metal powder into a burner flame, to thereby produce oxide ultra-microparticles, followed by solid-gas separation (see, for example, Patent Documents 11 to 16). There has also been proposed a method including spraying a gas to a molten metal, to thereby form a powder of the metal; conveying the powder by gas; and feeding the powder into a liquid where reaction such as chemical reaction or concentration is performed, thereby forming a micropowder (see patent Document 17). Furthermore, there

have been proposed methods for forming ultra-microparticles including applying plasma arc to a source such as bulk metal or a metal oxide rod, to thereby melt and evaporate the source, and spraying a reaction/cooling gas to the vaporized gas (see Patent Documents 18 to 20).

[0009]

However, the above dry-synthesis methods may be unsuitable for producing ITO powder. Thus, currently, dry-synthesis of ITO powder is not carried out on an industrial scale.

Patent Document 1: Japanese Patent Application Laid-Open (*kokai*) No. 62-21751

Patent Document 2: Japanese Patent Application Laid-Open (*kokai*) No. 5-193939

Patent Document 3: Japanese Patent Application Laid-Open (*kokai*) No. 6-191846

Patent Document 4: Japanese Patent Application Laid-Open (*kokai*) No. 2001-261336

Patent Document 5: Japanese Patent Application Laid-Open (*kokai*) No. 62-21751

Patent Document 6: Japanese Patent Application Laid-Open (*kokai*) No. 9-221322

Patent Document 7: Japanese Patent Application Laid-Open (*kokai*) No. 2000-281337

Patent Document 8: Japanese Patent Application Laid-Open (*kokai*) No. 2001-172018

Patent Document 9: Japanese Patent Application Laid-Open

(*kokai*) No. 2002-68744

Patent Document 10: Japanese Patent Application Laid-Open (*kokai*) No. 11-11946

Patent Document 11: Japanese Patent Publication (*kokoku*) No. 1-55201

Patent Document 12: Japanese Patent Publication (*kokoku*) No. 5-77601

Patent Document 13: Japanese Patent No. 3253338

Patent Document 14: Japanese Patent No. 3253339

Patent Document 15: Japanese Patent No. 3229353

Patent Document 16: Japanese Patent No. 3225073

Patent Document 17: Japanese Patent Application Laid-Open (*kokai*) No. 60-71037

Patent Document 18: Japanese Patent Application Laid-Open (*kokai*) No. 2002-253953

Patent Document 19: Japanese Patent Application Laid-Open (*kokai*) No. 2002-253954

Patent Document 20: Japanese Patent Application Laid-Open (*kokai*) No. 2002-263474

Disclosure of the Invention

Problems to be Solved by the Invention

[0010]

Under such circumstances, an object of the present invention is to provide a process for producing microparticles, which process enables production of microparticles such as oxide microparticles by means of a

simple apparatus at low cost and which is suitable for producing ITO powder. Another object of the invention is to provide an apparatus for producing the microparticles.

Means for Solving the Problems

[0011]

In a first mode of the present invention for attaining the aforementioned objects, there is provided a process for producing microparticles, characterized in that the process comprises feeding into a heat source a raw material in the form of a liquid stream, liquid droplets, or powder; capturing the formed product in the form of microparticles by means of a fluid of atomized liquid (hereinafter referred to as atomized liquid fluid); and collecting the microparticles in the form of slurry through gas-liquid separation.

[0012]

According to the first mode, the product obtained through feeding the raw material into the heat source is effectively captured in the form of microparticles by means of the atomized liquid fluid, and the microparticles are effectively collected in the form of slurry through gas-liquid separation.

[0013]

A second mode of the present invention is drawn to a specific embodiment of the process of the first mode, wherein the raw material to be fed into the heat source is provided through forming a molten material into a liquid stream or liquid droplets.

[0014]

According to the second mode, the raw material in the form of a liquid stream or liquid droplets formed from a molten material such as a metal or an alloy may be converted to an oxide thereof in the heat source, and the oxide can be captured in the form of microparticles by means of the atomized liquid fluid.

[0015]

A third mode of the present invention is drawn to a specific embodiment of the process of the first mode, wherein the raw material to be fed into the heat source is in the form of atomized powder.

[0016]

According to the third mode, the raw material in the form of atomized powder formed from a raw material such as a metal or an alloy is fed into the heat source, whereby the microparticles thereof are formed.

[0017]

A fourth mode of the present invention is drawn to a specific embodiment of the process of any of the first to third modes, wherein the gas-liquid separation is performed by means of a cyclone separator.

[0018]

According the fourth mode, the microparticles are effectively collected with the liquid fluid in the form of slurry through gas-liquid separation performed by means of a cyclone separator.

[0019]

A fifth mode of the present invention is drawn to a specific embodiment of the process of any of the first to fourth modes, wherein the heat source is acetylene flame or DC plasma flame.

[0020]

According to the fifth mode, the raw material is formed into microparticles thereof by acetylene flame or DC plasma flame.

[0021]

A sixth mode of the present invention is drawn to a specific embodiment of the process of any of the first to fifth modes, wherein the liquid fluid is water.

[0022]

According the sixth mode, the product is captured by water, and the product-water slurry is collected.

[0023]

A seventh mode of the present invention is drawn to a specific embodiment of the process of any of the first to sixth modes, wherein the raw material is at least one member selected from among metals, alloys, oxides, nitrides, and oxide nitrides.

[0024]

According the seventh mode, the raw material such as metal, alloy, oxide, nitride, or oxide nitride is formed into microparticles thereof.

[0025]

An eighth mode of the present invention is drawn to a specific embodiment of the process of any of the first to seventh modes, wherein the heat source is an oxidizing atmosphere or a nitrifying atmosphere, whereby oxide microparticles, nitride microparticles, or oxide nitride microparticles are produced.

[0026]

According to the eighth mode, the raw material is converted to oxide microparticles, nitride microparticles, or oxide nitride microparticles in an oxidizing atmosphere or a nitrifying atmosphere serving as a heat source.

[0027]

A ninth mode of the present invention is drawn to a specific embodiment of the process of any of the first to seventh modes, wherein the raw material is an In-Sn alloy or ITO powder, from which indium oxide-tin oxide powder is produced.

[0028]

According to the ninth mode, a slurry of ITO powder is produced from an In-Sn alloy or ITO powder.

[0029]

A tenth mode of the present invention is drawn to a specific embodiment of the process of the ninth mode, which process produces indium oxide-tin oxide powder having a tin content of 2.3 to 45 mass% as calculated on the basis of SnO_2.

[0030]

According to the tenth mode, the produced ITO maintains

conductivity by virtue of a predetermined amount of tin oxide.

[0031]

An eleventh mode of the present invention is drawn to a specific embodiment of the process of any of the first to tenth modes, wherein the product flows at a maximum speed of 150 m/sec or less, when the product is captured by means of the liquid fluid.

[0032]

According to the eleventh mode, microparticles can be produced at a relatively slow flow speed of the product.

[0033]

In a twelfth mode of the present invention, there is provided an apparatus for producing microparticles, characterized in that the apparatus comprises

an inlet for introducing, into the inside of the apparatus, a gas fluid and a product obtained through feeding a raw material in the form of a liquid flow, liquid droplets, or powder into a heat source;

a fluid jetting means for jetting an atomized liquid fluid to the introduced product;

a first gas-liquid separation means for subjecting, to gas-liquid separation, microparticles captured by the liquid fluid, to thereby form a slurry of the microparticles; and

a first circulating means for returning a part of an atmosphere fluid containing microparticles that have not been captured by the liquid fluid to a position where the fluid jetting means is disposed.

[0034]

According to the twelfth mode, the product obtained through feeding a raw material into a heat source is captured in the form of microparticles by means of an atomized liquid fluid, followed by gas-liquid separation, and at least a part of the atmosphere fluid is circulated through the circulating means, followed by another gas-liquid separation. Thus, the microparticles can be effectively collected.

[0035]

A thirteenth mode of the present invention is drawn to a specific embodiment of the apparatus of the twelfth mode, which apparatus further comprises, on the downstream side of the first gas-liquid separation means, a second gas-liquid separation means, the second gas-liquid separation means being provided for introducing a part of an atmosphere fluid containing microparticles that have not been captured by the liquid fluid, for jetting an atomized liquid fluid to the atmosphere fluid, and for performing gas-liquid separation, to thereby obtain a slurry of the microparticles.

[0036]

According to the thirteenth mode, the microparticles that have not been collected can be effectively collected through the second gas-liquid separation means.

[0037]

A fourteenth mode of the present invention is drawn to a specific embodiment of the apparatus of the thirteenth mode, which apparatus further comprises, on the downstream side of

the second gas-liquid separation means, a second circulating means for returning a part of an atmosphere fluid containing microparticles that have not been captured by the liquid fluid to the inlet of the second gas-liquid separation means.

[0038]

According to the fourteenth mode, the atmosphere gas which has not provided a slurry through the second gas-liquid separation means is further subjected to gas-liquid separation, whereby microparticles are effectively collected.

[0039]

A fifteenth mode of the present invention is drawn to a specific embodiment of the apparatus of any of the twelfth to fourteenth modes, wherein the first or second gas-liquid separation is a cyclone separator.

[0040]

According to the fifteenth mode, gas-liquid separation can be performed continuously and effectively by means of a cyclone separator.

[0041]

A sixteenth mode of the present invention is drawn to a specific embodiment of the apparatus of any of the twelfth to fifteenth modes, wherein the particles flow at a maximum speed of 150 m/sec or less, when the microparticles are captured by the liquid fluid jetted by means of the fluid jetting means.

[0042]

According to the sixteenth mode, microparticles can be

produced at a relatively slow flow speed.

Effects of the Invention

[0043]

As described hereinabove, according to the present invention, a raw material metal or alloy in the form of a liquid stream, liquid droplets, or powder is fed into a heat source, and the formed product in the form of microparticles is captured by means of an atomized liquid fluid. Thus, microparticles can be effectively produced in a simple manner.

Brief Description of the Drawings

[0044]

[FIG. 1] Schematic configuration of an embodiment of the apparatus of the present invention for producing microparticles.

[FIG. 2] An X-ray diffraction chart of ITO powder produced in Example 1 of the present invention.

[FIG. 3] An X-ray diffraction chart of ITO powder produced in Example 2 of the present invention.

[FIG. 4] An X-ray diffraction chart of ITO powder produced in Comparative Example 1 of the present invention.

[FIG. 5] An X-ray diffraction chart of ITO powder produced in Comparative Example 2 of the present invention.

[FIG. 6] An X-ray diffraction chart of ITO powder produced in Comparative Example 3 of the present invention.

[FIG. 7] An X-ray diffraction chart of ITO powder produced in Example 3 of the present invention.

[FIG. 8] An X-ray diffraction chart of ITO powder produced in Comparative Example 4 of the present invention.

Best Modes for Carrying Out the Invention

[0045]

According to the process of the present invention for producing microparticles, a raw material in the form of a liquid stream, liquid droplets, or powder is fed into a heat source.

[0046]

The raw material is, for example, metal or alloy, and specific examples include metals such as Mg, Al, Zr, Fe, Si, In, and Sn, and alloys thereof. The raw material may be any of the aforementioned oxides, nitrides, and oxide nitrides of the metal or alloy. As used herein, the "oxides" include compound oxides, and the "nitrides" include complex nitrides.

[0047]

The raw material to be fed may be melted to form a liquid stream or liquid droplets, or may be powder. In other words, a molten metal may be continuously poured from a tank in the form of a liquid stream or liquid droplets. Alternatively, the raw material to be fed may be formed into atomized powder.

[0048]

In the case where an In-Sn alloy is employed as a raw material, an ITO powder can be produced. Furthermore, when an ITO powder is employed as a raw material, a different type

of ITO material can be produced.

[0049]

The heat source may be an oxidizing atmosphere or a nitrifying atmosphere, and specific examples include acetylene flame and DC plasma flame. No particular limitation is imposed on the temperature of the heat source, so long as the heat source can melt metal, alloy, oxide, nitride, or oxide nitride and can sufficiently oxidize or nitrify the raw material. Conceivably, the temperature is at least some thousands of degrees Celsius in the case of acetylene flame, and at least some ten-thousands of degrees Celsius in the case of DC plasma flame.

[0050]

When a raw material in the form of a liquid stream, liquid droplets, or powder is fed into the above acetylene flame or DC plasma flame, a gas flow of a raw material itself, of the corresponding oxide, of the corresponding nitride, or of the corresponding oxide nitride is yielded as a product. The product may be a raw material itself (i.e., metal or alloy) or the corresponding oxide, nitride, or oxide nitride, depending on the flame conditions. In other words, when the flame is an oxidizing atmosphere, an oxide or oxide nitride of the metal or alloy is formed, whereas when the flame is a nitrifying atmosphere, a nitride or oxide nitride of the metal or alloy is formed. Alternatively, when an oxide, nitride, or oxide nitride is employed as a raw material, a different type of oxide, nitride, or oxide nitride may be

formed.

[0051]

According to the present invention, the formed product is captured by means of an atomized liquid fluid. Specifically, an atomized liquid fluid, preferably atomized water, is jetted to the product carried by a jet generated from the acetylene flame or DC plasma flame. By the mediation of the atomized liquid fluid, the product is quenched to form microparticles, and a slurry containing the microparticles in the jetted liquid is produced.

[0052]

No particular limitation is imposed on the type of the atomized liquid fluid to be fed, so long as the fluid can capture and cool the product. For example, when water is employed, water (preferably pure water) at ambient temperature is used. Alternatively, chilled water may also be used.

[0053]

The product flows at a maximum speed of, for example, 150 m/sec or less, preferably about 100 m/sec or less, when the product is captured in the form of microparticles.

[0054]

According to the present invention, the liquid fluid containing the microparticles captured by means of the jetted liquid fluid is subjected to gas-liquid separation, whereby the microparticles are collected in the form of slurry. No particular limitation is imposed on the method of collecting

the slurry, and a cyclone separator is preferably employed.

[0055]

According to the method of the present invention, when an In-Sn alloy or ITO powder is employed as a raw material, an indium oxide-tin oxide (ITO) powder can be produced. The thus-produced ITO powder contains a large amount of a SnO_2 solid solution component dissolved in In_2O_3. Therefore, the ITO exhibits high sinterability and readily provides high-density sintered ITO. As a result, a long-life sputtering target can be produced. When an ITO powder produced through any of various production methods or an ITO powder obtained by pulverizing sintered ITO is employed as a raw material, a different type of ITO powder having characteristics different from those of the raw material and containing a large amount of a SnO_2 solid solution component dissolved in In_2O_3 may be produced.

[0056]

The above ITO powder may be employed as a material for an ITO sputtering target. The ITO sputtering target material preferably has a tin content, as calculated on the basis of SnO_2, of 2.3 to 45 mass%.

Examples

[0057]

An embodiment of the apparatus for producing microparticles of the present invention will next be described with reference to FIG. 1.

[0058]

The apparatus has an inlet 10 for introducing, into the inside of the apparatus, a gas fluid and a product 3 obtained through feeding of a raw material 2 (e.g., metal or alloy) in the form of a liquid flow, liquid droplets, or powder into a flame 1 (acetylene flame or DC plasma flame) serving as a heat source that can provide an oxidizing atmosphere or a nitrifying atmosphere; fluid jetting means 20 for jetting an atomized liquid fluid to the introduced microparticles; a cyclone separator 30 serving as gas-liquid separation means for subjecting, to gas-liquid separation, the microparticles captured by the liquid fluid, to thereby form a slurry of the microparticles; and circulating means 40 for returning a part of an atmosphere fluid containing microparticles that have not been captured by the liquid fluid to a position where the fluid jetting means is disposed.

[0059]

No particular limitation is imposed on the type of the inlet 10, so long as the inlet allows a gas flow containing a product to feed into the inside of the apparatus. The inlet may be gas-suction means.

[0060]

The fluid jetting means 20 is provided in a conduit 11 on the downstream side of the inlet 10. The fluid jetting means 20 includes, for example, a plurality of jet nozzles 21 for jetting water, a pump 22 for feeding fluid to the jet nozzles 21, and a fluid tank 23 for storing fluid. No particular limitation is imposed on the jetting direction of

the fluid jetted through the jet nozzles 21. However, the jetting direction is preferably such that the jetted fluid is merged with a gas flow introduced through the inlet 10. The product 3 contained in the gas fluid introduced through the inlet 10 is cooled by means of the atomized liquid fluid (e.g., water) to form microparticles, and the microparticles are captured. In the conduit 11, a venturi section 12, where the flow path is narrowed, is provided on the downstream side of the jet nozzles 21, so as to prevent reduction in flow rate of a gas-liquid mixture. Provision of the venturi section 12 is not obligatory. The jet nozzles 21 and the pump 22 are not necessarily provided, and instead, the liquid may be jetted on the basis of suction power generated by flow of gas.

[0061]

The conduit 11 provided with the inlet 10 is in communication with an inlet 31 of the cyclone separator 30 serving as gas-liquid separation means. A gas-liquid mixture which has been introduced through the inlet 31 into the cyclone separator 30 forms a vortex 33 proceeding around the inner wall of a cyclone body 32, whereby a liquid component is separated from the gas. The liquid component; i.e., a slurry containing the microparticles, falls down in the cyclone separator 30, and a gas component is discharged through a gas-discharge outlet 34.

[0062]

In the apparatus of the embodiment, the circulating

means 40 is provided so as to communicate with the gas-discharge outlet 34. In other words, circulation piping 41 is connected to the outlet 34, the circulation piping 41 being in communication with a position near the inlet 10 of the conduit 11. A blower 42 intervenes in the circulation piping 41. The circulation means 40 consists of the members 41 and 42. Through the circulation means 40, the powder which has not been captured is returned to the upstream side of the jet nozzles 21, thereby enhancing capturing efficiency.

[0063]

The liquid component which has been separated from the gas by means of the cyclone separator 30 is discharged through a water-discharge outlet 36 and stored in the fluid tank 23. The supernatant water of the slurry stored in the tank 23 is circulated by means of the circulation means 40, whereby the concentration of the slurry containing the microparticles gradually increases.

[0064]

Most of the discharged gas produced by means of the cyclone separator 30 is circulated through the gas-discharge outlet 34 to the circulation piping 41. A part of the discharged gas; for example, about 1/10 of the amount of the discharge gas, is discharged through a second gas-discharge outlet 35.

[0065]

In the apparatus of the present embodiment, a second cyclone separator 50 serving as second gas-liquid separation

means is connected to the second gas-discharge outlet 35 via discharge piping 43. The second cyclone separator 50 has virtually the same structure as the cyclone separator 30 and serves as gas-liquid separation means. Specifically, a gas-liquid mixture which has been introduced through an inlet 51 connected to the discharge piping 43 into the second cyclone separator 50 forms a vortex 53 proceeding around the inner wall of a cyclone body 52, whereby a liquid component is separated from the gas. The liquid component; i.e., a slurry containing the microparticles, falls down in the cyclone separator 50, and is discharged through a water-discharge outlet 54 and stored in a fluid tank 61. More specifically, a venturi section 44, where the flow path is narrowed, intervenes in the discharge piping 43, and water circulating piping 62 is provided so as to maintain communication between the venturi section 44 and the fluid tank 61. When high-speed gas flow is provided in the venturi section 44, water contained in the fluid tank 61 is drawn and jetted into the venturi section 44, whereby microparticles remaining in the gas phase are captured by water (liquid). Gas-discharge piping 71 is connected to a gas-discharge outlet 55, and a second blower 72 is provided in the gas-discharge piping 71, such that the gas is discharged through the gas-discharge outlet 55 by the mediation of the second blower 72. Water contained in the water tank 61 may be jetted into the gas-discharge piping 43 by means of a pump and jet nozzles as mentioned in relation to the cyclone separator 30. As also

mentioned above, the fluid tank 61 may be provided with a filter and a settling tank for separating microparticles from the liquid through neutralization. In addition, a portion of the gas discharged through the gas-discharge outlet 55 may be circulated to the upstream side of the venturi section 44 of the gas-discharge piping 43, to thereby enhance capturing efficiency.

[0066]

When the cyclone separator 30 provides sufficient microparticle-capturing efficiency, the second cyclone separator 50 is not necessarily provided. In order to further enhance capturing efficiency, a plurality of cyclone separators may be linked together.

[0067]

Production Examples of microparticles by means of the apparatus of the above embodiment will next be described.

[0068]

<Example 1>

An atomized powder (mean particle size: 45 μm) of In-Sn alloy (Sn: 9.6 wt.%) was introduced to acetylene flame, to thereby synthesize an ITO (In_2O_3:SnO_2 = 90 : 10 wt.%) powder under dry conditions. The powder was collected by means of a bag filter under dry conditions, to thereby yield an ITO powder of Example 1.

[0069]

<Example 2>

In a manner similar to that of Example 1, an ITO powder

was synthesized by means of acetylene flame under dry conditions. The powder was collected by jetting water to the powder under wet conditions, to thereby yield an ITO powder of Example 2.

[0070]

<Comparative Example 1>

An indium oxide powder which had been synthesized under wet conditions was calcined at 1,000°C. Similarly, a tin oxide powder which had been synthesized under wet conditions was calcined at 1,000°C. The thus-calcined indium oxide powder (90 mass%) and tin oxide powder (10 mass%) were mixed by means of a mortar, to thereby yield an oxide powder of Comparative Example 1 (Standard Product 1).

[0071]

<Comparative Example 2>

An ITO powder was synthesized through co-precipitation under wet conditions, to thereby yield an ITO powder of Comparative Example 2.

[0072]

The co-precipitation wet synthesis was performed through the following procedure. First, In (4N) (20 g) was dissolved in nitric acid (special-grade reagent, concentration: 60 to 61%) (133 cc) at ambient temperature, to thereby obtain a solution (pH = -1.5). Similarly, Sn (4N) (2.12 g) was dissolved in hydrochloric acid (special-grade reagent, concentration: 35 to 36%) (100 cc) at ambient temperature, to thereby obtain a solution (pH = -1.9). The

two solutions were mixed, to thereby obtain a mixed-acid solution. No precipitation was observed during mixing, and the mixed solution was found to have a pH of -1.5. Subsequently, 25% aqueous ammonia (special-grade reagent) was added to the acidic solution for neutralization, to thereby adjust the pH to 6.5, whereby a white matter was precipitated. Several hours after, the supernatant was removed, and the precipitate was washed with pure water (2L × 3), followed by drying at 80°C, roasting at 600°C for three hours, and dehydration, to thereby yield an ITO powder through wet synthesis.

[0073]

<Comparative Example 3>

A mixture (tin oxide content: 10 wt.%) of an indium oxide powder and a tin oxide powder which had been synthesized under wet conditions was sintered at 1,550°C or higher. The sintered ITO was pulverized, to thereby yield an ITO powder of Comparative Example 3.

[0074]

<Test Example 1>

Each of the ITO powders of Examples 1 and 2 and Comparative Examples 1 to 3 was analyzed in terms of SnO_2 solid solution content. The determination procedure was as follows. Prior to the test, ITO powders of Examples 1 and 2 and Comparative Examples 2 and 3 were calcined at 1,000°C for three hours in air so as to grow precipitated SnO_2 microparticles to SnO_2 large particles, which are readily

detectable.

1. Inductively coupled high-frequency plasma spectroscopic analysis (ICP spectroscopic analysis) was performed. For calculation, it was assumed that each ITO powder exclusively consists of In, Sn, and oxygen (O), and that a certain amount of oxygen may be deficient. The ratio of In to Sn was calculated from the analytical values, and the ratio by weight of In_2O_3 to SnO_2 was calculated, under the condition that all elemental In and Sn were converted to In_2O_3 and SnO_2, respectively.

2. ITO powders of Examples 1 and 2 and Comparative Examples 1 to 3 were subjected to powder X-ray diffractometry (XRD: by means of MXP 18II, product of Mac Science), whereby the precipitated SnO_2 content of each powder was determined. In each case, the presence of a compound oxide ($In_4Sn_3O_{12}$) was checked from the corresponding diffraction chart. When the compound oxide was not detected, the precipitated SnO_2 content (mass%) of the ITO powder was determined from the ratio between integral diffraction intensity attributed to In_2O_3 (222) and integral diffraction intensity attributed to SnO_2 (110), with respect to Standard Product 1 of Comparative Example 1. Specifically, the precipitated SnO_2 content (mass%) is a SnO_2 content obtained from an integral intensity of X-ray diffraction attributed to SnO_2, assuming that the SnO_2 component which has not been dissolved in In_2O_3 and has been grown through calcination at about 1,000°C exhibits an X-ray diffraction peak attributed to SnO_2 (110). FIGs. 2 to

6 show the results of X-ray diffraction analysis.

3. On the basis of the results of "1." and "2.," the SnO_2 solid solution (in In_2O_3) content of each ITO powder was obtained from the amount of SnO_2 which had been detected through ICP analysis but which had not been detected as SnO_2 (110) through X-ray diffraction.

[0075]

The results are shown in Table 1.

[0076]

The ITO powders of Examples 1 and 2 were found to have a SnO_2 solid solution content of 2.35 wt.% and 2.42 wt.%, which are higher than the SnO_2 solid solution content of 2.26 wt.% of the ITO powder of Comparative Example 2 obtained through wet synthesis. The ITO powder of Comparative Example 3, which had been produced through pulverizing the sintered product thereof, was found to form a compound oxide. Therefore, the SnO_2 solid solution content of the ITO powder of Comparative Example 3 could not be determined.

[0077]

[Table 1]

Sample No.	ICP analysis				XRD analysis				SnO_2 solid solution content (wt.%)
	In (wt.%)	Sn (wt.%)	In_2O_3 (wt.%)	SnO_2 (wt.%)	Compound oxide	InO_3 (222)	SnO_2 (110)	Precipitated SnO_2 content (wt.%)	
Ex. 1	74.1	8.26	89.52	10.48	no	6974596	357821	8.13	2.35
Ex. 2	74.8	7.90	89.92	10.08	no	6875331	331124	7.66	2.42
Comp. Ex. 1	75.1	7.87	90.09	9.91	no	7141621	455777	9.91 standard	0.00
Comp. Ex. 2	76.1	8.03	90.03	9.97	no	7273411	352429	7.71	2.26
Comp. Ex. 3	74.8	7.90	90.02	9.98	yes	7529677	105639	—	—

[0078]

<Example 3>

An atomized powder (mean particle size: 45 μm) of In-Sn alloy (Sn: 9.6 wt.%) was introduced to DC plasma flame, to thereby synthesize an ITO (In_2O_3:SnO_2 = 90 : 10 wt.%) powder under dry conditions. The powder was collected by jetting water to the powder under wet conditions, to thereby yield an ITO powder of Example 3.

[0079]

<Comparative Example 4>

Similar to Comparative Example 1, an indium oxide powder which had been synthesized under wet conditions was calcined at 1,000°C. Similarly, a tin oxide powder which had been synthesized under wet conditions was calcined at 1,000°C. The thus-calcined indium oxide powder (90 mass%) and tin oxide powder (10 mass%) were mixed by means of a mortar, to thereby yield an oxide powder of Comparative Example 4 (Standard Product 2).

[0080]

<Test Example 2>

Similar to Test Example 1, each of the ITO powders of Example 3 and Comparative Example 4 was analyzed in terms of SnO_2 solid solution content. Powder X-ray diffractometry (XRD) was performed by means of X'PertPRO MPD (product of Spectris Co., Ltd.). The results are shown in Table 2. FIGs. 7 and 8 show the results of X-ray diffraction analysis.

[0081]

The ITO powder of Example 3 was found to have a SnO_2 solid solution content of 3.00 wt.%, which is remarkably higher than the SnO_2 solid solution content of the ITO powder of Example 2 obtained by means of acetylene flame instead of DC plasma flame.

[0082]

[Table 2]

Sample No.	ICP analysis				XRD analysis				SnO_2 solid solution content (wt.%)
	In (wt.%)	Sn (wt.%)	In_2O_3 (Wt.%)	SnO_2 (Wt.%)	Compound oxide	InO_3 (222)	SnO_2 (110)	Precipitated SnO_2 content (wt.%)	
Ex. 3	73.8	7.46	90.40	9.60	no	691582	31090	6.60	3.00
Comp. Ex. 4	75.1	7.86	90.10	9.90	no	892303	62325	9.90	0.00